

DESIGNING OF CONTINUOUS FILTRATION AND DRYING OPERATIONS CAPABLE FOR WORKING OUT EFFECTIVELY IRRESPECTIVE OF FLOW RATES AND CONDITIONS WITHOUT VARYING THE SIZING OF FILTRATION AND DRYING EQUIPMENTS

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ABSTRACT

As the flow chemistry is an emerging field in chemical industries in near future and hence there is a need to design the chemical engineering unit operations like filtration and drying which can work in a continuous manner. The designed system should be capable for working out effectively at various flow rates under different conditions without varying the sizing of equipments. The rate of filtration depends upon the size of solid particles. As the size of solid particles increases then the voidage of filtered cake increases and the cake resistance decreases and this finally increases the rate of filtration. As the size of solid particles increases then the surface area per unit mass decreases and hence the overall quantity of the moisture going to adsorb on the surface of particles decreases. Therefore, the required heat duty for carrying out the drying operation reduces. As the size of solid particles increases then the voidage between them increases and the resistance to the flow of evaporating moisture going to release from the solid mass decreases while drying operation is in progress. The size of particles forming during crystallization or precipitation depends upon the cooling rate and hydrodynamic conditions maintained while carrying out the crystallization or simple precipitation or reactive precipitation. By considering all these technical aspects, the system of carrying out continuous filtration and drying operation has been designed in such a way that it is capable of working out effectively irrespective of flow rates within the acceptable range and at different conditions affecting the size of particles without varying the size of filtration and drying unit. The designed system has been validated for carrying out filtration and drying of the precipitated calcium oxalate formed from the reaction of calcium nitrate with oxalic acid in aqueous condition.

Keywords: *filtration, drying, continuous process, crystallization, precipitation etc.*

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1. INTRODUCTION

1.1 Filtration

Filtration is one of the unit operations of chemical engineering in which solid particles can be removed from the fluid by passing the fluid through a filtering medium or septum, on which the solids get deposit. The fluid may be liquid or a gas; the valuable stream of the filtration may be the fluid, or both. Sometimes it is neither because filtration is implemented just to treat out the discarded waste stream of chemical industries (McCabe and Smith; 1993). Pressure difference across the filter medium is necessary to carry out the filtration operation. Filters are generally classified into two types. First operates by maintaining the pressure above atmospheric on the up-stream side of the filter medium and second which operates with atmospheric pressure on the upstream side and a vacuum on the downward side of the filter medium (McCabe and Smith; 1993). These filters are further classified into two main groups: Clarifying filters and cake filters. Clarifying filters are useful to remove small amounts of solids to

produce a clean gas or a clear liquid. Cake filters are useful for carrying out the separation of larger amounts of solids in the form of cake of crystals or sludge. They include the provision for creating the pressure drop across the filter medium for removing as much residual liquid from the solids as possible before discharge (McCabe and Smith; 1993). In such type of filtration, the cake resistance is relatively higher as compared to filter medium resistance. At the start of filtration some solid particles enter inside the pores of filter medium and get immobilised. But later on start to collect on the filter medium surface. If the filtration is carried out in a batch mode then after the brief initial period the cake of solids does the filtration and not the septum or filter medium. These cake filters may operate by pressurising the upstream side above than the atmospheric pressure or with vacuum applied below the surface of filter medium. Therefore, cake filters are further classified into two types as pressure filters and vacuum filters. Both these types can be operated in a continuous or batch wise manner. If it is essential to avoid the contamination of impurities going to deposit on the filtered solid mass above the filter medium then the upstream side should be enclosed and operate at higher pressure than the atmospheric above the upstream side and in a batch mode. The second scientific or technical reason of operating the pressure filters in a batch mode is that the continuous discharge of solids against the positive pressure becomes difficult. Therefore, vacuum filters are generally used for carrying out the filtration in a continuous manner (McCabe and Smith; 1993).

1.2 Drying

Drying is also one of the unit operations of chemical engineering and it is generally carried out after the filtration. Moisture associated with the wet filtered cake can be removed by carrying out the drying operation by increasing the temperature of solid mass. Drying is suitable for removing a relatively small amount of water or other liquid from the solid mass and reduces the content of residual liquid to an acceptable low value which is called as an equilibrium moisture content. Equilibrium moisture content depends upon the temperature at which drying is carried out and it also depends upon the humidity or the content of other liquid (to be filtered out) in the surrounding atmosphere of the drying solid mass (McCabe and Smith; 1993). There are two types of dryers as a continuous and batch wise. These dryers are further classified as direct and indirect. Direct dryers are those in which the solid mass to be drying out is directly exposed to a hot gas or air. Indirect dryers are those in which the heat is going to transferred to the solid to be drying out through a metal surface with which solid is in contact (McCabe and Smith; 1993). Drying operation takes place in two parts. First part is that in which moisture from the solid gets removed at the constant drying rate and it is called as constant drying rate period. Once the constant drying rate period ends then the drying rate falls down. The moisture content inside the solid mass at which drying rate falls down is called as a critical moisture content. The critical moisture content is the property of the solid to be dried out because the affinity of getting adsorb of moisture on different solids varies from solid to solid. Further it also depends upon the thickness of the material, the rate of drying and the resistance to the heat and mass transport of the water or liquid vapours within the solid (McCabe and Smith; 1993). Wet solid contains free moisture content which is having same vapour pressure as that of continuous pool of liquid at the same temperature.

Carrying out filtration and drying operation in a continuous manner is comparatively more difficult than carrying out in the batch mode. As the flow chemistry is an emerging field in chemical industries in near future then it is an essential to develop or design the continuous system of filtration and drying in such a way that the designed units should capable for working out effectively at various flow rates within the acceptable range and at different conditions affecting the particle size without varying the sizing of filtration and drying units.

2. THE SELECTION OF SUITABLE SYSTEM

The system chosen is to carry out the filtration and of hydrated calcium oxalate from water and then drying is necessary to remove the moisture contents from filtered wet cake of calcium oxalate. Calcium oxalate can be prepared by carrying out the reaction of calcium nitrate with oxalic acid in aqueous condition. As calcium nitrate reacts with oxalic acid then it produces calcium oxalate as follow:



As the reaction between calcium nitrate and oxalic acid is of ion exchange type of instantaneous in nature and formed calcium oxalate having negligible solubility in water then it precipitate out immediately and hence it is called reactive precipitation. The chosen molar concentrations of calcium nitrate and oxalic acid are of 0.1 mol/lit and 0.5 mol/lit respectively. The reaction has been carried out initially in a batch reactor in order to produce the slurry of calcium oxalate in water. The resulted slurry has been used to find out the particle size distribution. The same slurry of calcium oxalate in water has been used to carry out the filtration study in order to estimate the filter medium resistance and specific cake resistance. The filtered wet solid mass has been used to find out the drying kinetics such as constant drying rate, critical moisture content and equilibrium moisture content. All these parameters estimated from the batch studies have been used to design the continuous filtration and drying system.

3. ESTIMATION OF THE PARTICLE SIZE

As the particle size increases then the voidage of the filtered cake increases (Zheng Yang et al; 2005). As the voidage increases, then the resistance to the flow of filtrate through the filtered cake decreases and filtration rate increases (Stevenson; 1997). As the size of particle increases, then the surface area per unit mass decreases and therefore the entire quantity of moisture going to adsorb on solid surface decreases. Therefore, heat duty required for carrying out drying operation reduces. As the voidage of solid bed increases then the resistance to the flow of evaporated moisture (through drying operation) through the solid bed decreases and evaporated moisture gets escape from the solid bed to the surrounding atmosphere at faster rate. It means that drying rate increases with increase in particle size. Therefore, it is concluded that the rate of filtration and drying operation depend upon the size of solid particles. Therefore, it is an essential to estimate the mean diameter of solid particle and particle size distribution before going to carry out the designing of filtration and drying operations. The solid particles of calcium oxalate going to generate into the system under consideration via reactive precipitation carried out by reacting calcium nitrate with oxalic acid in aqueous condition. As the reaction between calcium nitrate and oxalic acid is of an ion exchange type of reaction and hence it is an instantaneous type of reaction. As the reaction is an instantaneous in nature and the formed solid product calcium oxalate having negligible solubility in water, the formed solid gets precipitate out instantly. The rate of nuclei generation is very high and these generated nuclei are not getting sufficient time to arrange themselves in a specific order to form a particular shape. Therefore, these nuclei going to agglomerate randomly and formed powder is completely amorphous in nature. Therefore, it is assumed that the solid particles formed from the random agglomeration of nuclei are spherical in shape.

3.1 Experimental Set-up

Experimental set-up as shown in Figure-1 was used to carry out the reaction between calcium nitrate and oxalic acid in aqueous condition in order to produce the calcium oxalate. The experimental set-up as shown in Figure-1 consists of a batch reactor of having the reaction volume of 600 ml. The height of liquid (H) and diameter of reactor (T) are of equal

size of 0.09 m. Diameter of the pitched blade stirrer (D), width of the baffle (W) and height of the stirrer from the bottom of the reactor (h) should be equal to $T/3$, $T/12$ and $T/3$ respectively as per the standard designing of mechanical agitated contractor (McCabe and Smith; 1993). The dimensional values of D, W and H are equal to 0.03 m, 0.0075 m and 0.03 m respectively.

3.2 Experimental Procedure

500 ml of 0.1 M of aqueous calcium nitrate solution taken into the reactor and then stirring was initiated at the stirring speed of 500 RPM. Then 100 ml of 0.5 M of aqueous oxalic acid solution (Stoichiometric amount) was charged to the reactor and stirred it for 15 minutes which is the typical time period to complete the reaction. Once the stirring is in continuation after charging both reactant then after 15 to 20 minutes of time interval, the sample of 2.5 ml was collected and injected into the Beckman Coulter Counter in order to find out mean diameter of solid particles and range of diameter of different particles. Same experiment was performed at different speed of agitation in order to know the effect of stirring speed on mean particle diameter.

3.3 RESULTS AND DISCUSSION

3.3.1 Variation in mean particle diameter with time

The mean particle diameter was measured with the help of Coulter Counter and the variation in mean particle diameter with time has been shown graphically in Figure-2. From the Figure-2, it is cleared that mean particle diameter is increasing within first 30 minutes from $6.549\ \mu\text{m}$ to $9.049\ \mu\text{m}$ and later on it remains almost same.

3.3.2 Effect of stirring speed on the mean particle diameter

The above experiment has been performed at various speed of agitation and the mean particle diameter achieved in first 15 minutes (which is typically reaction period) was measured with the help of Coulter Counter. The variation in mean particle diameter with speed of agitation has been shown in Figure-3. From the Figure-3, it is cleared that as the speed of agitation increases from 0 to 100 RPM then the particle size also increases because nuclei or particles should remain in suspension for the growth and keep getting agglomerated. But any further increase in speed of agitation results into the reduction of particle size and this may be due to high shear generated at high speed of agitation and resulting into particle attrition.

3.3.3 Estimation of particle size distribution

The particle size distribution has been estimated as follow. The data noted down in Table-1 has been obtained from the Coulter Counter.

Table 1: Volume % of Solid Having Different Range of Particle Size

Range of particle diameter (μm)	Volume % of solid	Volume of the sample injected in Coulter Counter (ml)	Obscuration or Vol% of solid in the injected sample
0 - 0.375	0.068	2.5	11
0.375 - 0.412	0.122		
0.412 - 0.452	0.178		
0.452 - 0.55	0.25		

The volume of injected sample in Coulter Counter is of 2.5 ml. From the Coulter Counter data it has been known that the volume of solid in the injected sample is of 11%. Therefore, the entire volume of solid injected in the Coulter Counter is of 0.275 ml. The mean diameter of the solid particles having the diameter in the range of 0 - 0.375 μm is of 0.1875 μm . The solid particles having the diameter in the range of 0 - 0.375 μm occupies the volume of 0.068 % of entire solid injected in the Coulter Counter. Therefore, the volume of solid particles having the diameter in the range of 0 - 0.375 μm is of 0.000187 ml. The volume of one particle has been estimated by knowing the mean diameter and it is of 3.45×10^{-15} ml. The number of particles having the diameter in the range of 0 - 0.375 μm has been estimated by knowing the volume of all solid particles of having the diameter in the range of 0 - 0.375 μm and the volume of one particle. The estimated number of particles having the diameter of particles in the range of 0 - 0.375 μm is of around 5.4×10^{10} . In such a way the number of particles having the diameter in various ranges has been estimated and resulted particle size distribution has been shown in Figure-4.

4. STUDY OF THE FILTRATION OPERATION

Filtration is required to separate out solid from liquid or gas by means of filtering medium, which retains solids on the filter media and allow fluid to pass through it. Filtration flux depends upon the resistance offered by the filter media as well as solid cake. The flow of the filtrate through the cake and filter media can be related to the pressure drop across the filter media by means of Ergun equation (McCabe and Smith; 1993).

$$\frac{t}{V} = \left(\frac{\mu}{A\Delta P} \right) X \left(\frac{\alpha C V}{2A} + R_m \right) \quad (2)$$

The above equation (2) can be simplified as follow:

$$\frac{t}{V} = B_1 X V + B_2 \quad (3)$$

Where, and

$$B_1 = \frac{\mu \alpha C}{2A \cdot A \cdot \Delta P} \quad B_2 = \frac{\mu R_m}{A \Delta P}$$

Where, α is the specific cake resistance (m/Kg), R_m is the filter medium resistance (m^{-1}), μ is the viscosity of filtrate (Pa.s), C is the concentration of slurry (Kg/m^3), A is the area of filter (m^2) and ΔP is the pressure drop across the filter media (N/m^2). Specific cake resistance has been estimated by carrying out the batch filtration experiment.

4.1 Experimental Set-up

The experimental set-up of batch filtration unit has been shown in Figure-5.

4.2 Experimental Procedure

Slurry of calcium oxalate monohydrate in water having the concentration of 12 kg/m^3 was prepared by carrying out the reaction of 0.1 molar of calcium nitrate with 0.5 molar of 50% excess oxalic acid. System as shown in Figure-5 was kept ready by applying the vacuum. Well stirred slurry was charged continuously into the Buchner funnel and simultaneously stop watch was started. Time required for achieving different quantity of filtrate going to accumulate inside the vacuum flask was noted down in Table-2.

4.3 Estimation of specific cake resistance and the filtration area of the continuous Belt filter

Table 2: Observations of Filtration Experiment

Concentration of slurry (12 kg/m ³)		
Volume of filtrate collected (V) (ml)	Time (sec)	t/V (sec/m ³)
100	6	60000
200	32	160000
300	61	203333.33
400	114	285000
500	171	342000
600	237	395000
700	299	427142.86
800	361	451250
900	428	475555.56
1000	499	499000

The plot of t/V Vs. V has been shown in Figure-6. Plot of t/V Vs. V gives the slope (BI) equal to 5×10^8 (s/m⁶). Further by knowing the value of BI , area of the filter (A) and all other parameters like pressure drop of 10 mmHg, viscosity of the filtrate of about 10^{-3} Pa.s, the value of specific cake resistance has been estimated and it is of around 3.19×10^9 (m/kg). The flow of filtrate to filter out is of around 4.23×10^{-5} m³/s having the slurry concentration of calcium oxalate monohydrate of 12 kg/m³. By knowing the flow rate, concentration of solid inside the slurry and by neglecting the filter medium resistance and the area of belt filter required for carrying out filtration has been estimated and it is of around 25 cm². It was assumed that the rate of filtration remains same throughout the filtration operation because it was carried out in a continuous manner and the rate of filtration was assumed same as the input feed rate of mother liquor of the belt filter. Therefore, filtration takes place within first 5 seconds once the feed of mother liquor reaches on the filter media of belt filter. It was assumed that the filtered cake having the residence time of 20 seconds on the surface of filter media of the belt filter and therefore the belt filter of having the upstream surface area of 500 cm² was fabricated. By knowing the upstream surface area of belt filter, the dimensions of the belt filter have been estimated. The fabricated belt filter was 40 cm in length and 12.5 cm in width.

4. STUDY OF DRYING KINETICS

Drying is carried out after the filtration. Drying is necessary for the removal of moisture from wet solid.

4.1 Experimental Procedure

64 gm of wet solid was taken into the petty dish. Then the petty dish containing the sample was kept inside the oven at 100°C. The weight of the sample in petty dish was measured after every 2 minutes of time interval. After one hour of time interval, the sample was kept for drying for three days continuously specifically in dry atmosphere till the constant weight

was obtained. As the atmosphere is completely dry then it can be concluded that there will be no any equilibrium moisture content after three days of drying and the solid mass was considered as completely free of moisture.

4.2 Estimation of the drying rate

The graph of drying rate obtained at 100°C has been shown in Figure - 7. From this graph it has been observed that 1.2 and 0.2 are the initial and critical moisture content per gm of dry solid respectively and 35 gm/min m² is the constant drying rate. The area required for drying has been estimated as follow (McCabe and Smith; 1993):

$$A = \frac{M(X_i - X_c)}{t_c R_c} \quad (4)$$

Where, X_i and X_c are the initial and critical moisture content per unit mass of dry solid, M is the mass of dry solid R_c is the constant drying rate and t_c is the constant drying rate period. The dry solid mass exits the filtration unit at the rate of 30 gm/min along with 1.2 gm of water moisture per gram of dry solid and fed to the dryer. The residence time of the material inside the screw conveyor dryer was assumed to be 10 minutes for carrying out the sizing of continuous screw conveyor dryer. Therefore, the estimated area of 860 cm² from the equation (4) has to sweep by the wet material entering the screw conveyor dryer in order to reduce the moisture content from its initial value of 1.2 to the critical value of 0.2.

4.3 Sizing of the screw conveyor dryer

The schematic of screw conveyor dryer has been shown in Figure-8. The cross-sectional view of the dryer has been shown in Figure-9. As the flow rate of dry solid mass and moisture content along with the dry solid is known then the volumetric flow rate of wet solid going to feed to the dryer has been estimated by adding the volume of dry solid and moisture content together. Even though the volume is not additive property but the volume of solid mass and moisture content added together in order to find out the maximum volume going to occupy by the incoming feed of wet solid inside the screw conveyor dryer. The volume swept by the incoming material inside the screw conveyor dryer is given by

$$\left(\frac{\pi D^2 \theta}{8 \times 3.14} - 0.25 * \left(1 - \left(\sin \left(\frac{\theta}{2} \right) \right)^2 \right)^{0.5} \sin \left(\frac{\theta}{2} \right) \right) L = Q * t = 497 \text{ ml} \quad (5)$$

The area swept by the incoming material inside the screw conveyor dryer is nothing but the area required for drying which has been already estimated and it is equal to 860 cm². This drying area is given by

$$\frac{\pi D \theta L}{6.28} = 860 \quad (6)$$

The diameter of screw was assumed to be 10 cm and clearance between screw and inner wall of the covering pipe was assumed to be 0.2 cm and therefore inner diameter of covering pipe (D) to be equal to 10.4 cm. The diameter of shaft was assumed to be 2.1 cm. The values of θ and L has been estimated by solving the equations (5) and (6) simultaneously and θ equal to be 1.14 radian and length of screw conveyor dryer (L) equal to be 144 cm. The fabricated screw conveyor dryer was having the inner diameter (D) of 10.4 cm and length (L) of 144 cm.

5. CONFIGURATION OF FILTRATION AND DRYING OPERATIONS

The schematic of continuous filtration operation has been shown in Figure-10. It consists of belt filter. Vacuum trap system of belt filter has been shown in Figure-10. As vacuum applied then the pressure inside the drainage pipe get reduces.

Therefore, once the vacuum inside the drainage pipe gets develop then the pressure inside the drainage pipe (P_A) becomes less than the outside atmospheric pressure (P_B). The drainage pipe was dipped in the water taken into the outer container-1 which was open to the atmosphere. The sufficient clearance was maintained between the bottommost ending point of drainage pipe and the bottom of outer container-1 so that the filtrate accumulated inside the drainage pipe gets flow easily from the drainage pipe to the outer container-1. The filtrate was going to take out from the outer container-1 through the pipe-2 as a overflow. As the vacuum inside the drainage pipe gets developed then the height of liquid inside the drainage pipe gets reached more than the liquid height inside the outer container-1 and the pressure gets balance as follow:

$$P_A + \rho gH = P_B \quad (7)$$

Once the height of the filtrate accumulated inside the drainage pipe reached above than the height H then according to Bernoulli's theorem, liquid started to flow from the drainage pipe to the outer container-1 and finally left the system as a overflow through the pipe-2.

Vacuum adjustable valve was configured to adjust the vacuum inside the vacuum trap system. If the opening of the vacuum adjustable valve increases then the vacuum strength inside filter tray get reduces and vice-versa. If the input flow rate of mother liquor to the belt filter increases then it has to filter out higher rate. As the rate of filtration depends upon the pressure drop across the filter medium then the rate of filtration can be increased by increasing the pressure drop across the filter medium. The pressure drop across the filter medium can be increased by increasing the vacuum strength inside the filter tray and it can be achieved by reducing the opening of vacuum adjustable valve. As already discussed, the rate of filtration depends upon the size of solid particles and it increases with increase in size of particles because of more voidage going to develop inside the filtered cake due to larger particle size. As the particle size decreases then the rate of filtration get reduces and in such a case the rate of filtration can be increased by increasing the pressure drop across the filter media. The pressure drop across the filter media can be increased by increasing the strength of vacuum inside the filter tray and it can be achieved by reducing the opening of vacuum adjustable valve. The designed belt filter was operated successfully at various flow rates and various particle sizes but the variation should be within the acceptable range.

The schematic of continuous drying operation has been shown in Figure-11. As the drying section is considered, the drying rate increases with increase in particle size because the voidage going to develop inside the solid bed is more for a comparatively larger particle size and therefore escaped moisture get easily moved out from the solid bed. One more scientific concept is that as the particle size increases then the surface area per unit mass decreases. Therefore, the entire quantity of moisture going to adsorb on the surface of solid particles decreases and the heat duty required for carrying out overall drying operation reduces. If the particle size is comparatively less then more compact solid bed gets form and drying rate get reduces. Therefore, the drying rate depends upon the size of solid particles. If the solid material having comparatively smaller particle size gets entered inside the screw conveyor dryer then the rate of drying operation can be increased by increasing the strength of vacuum inside the screw conveyor dryer. It can be achieved by reducing the opening of vacuum adjustable valve. If the flow rate of wet solid mass to the continuous screw conveyor dryer increases then the temperature of solid mass is not going to reach to the temperature at which acceptable drying rate can be achieved. In such a case the boiling point of water has to reduce by increasing the strength of vacuum inside the screw conveyor dryer and it can be achieved by reducing the opening of vacuum adjustable valve. The designed dryer was operated successfully at various flow rates and various particle sizes but the variation should be within acceptable range.

CONCLUSION

The mean diameter of particles of calcium oxalate monohydrate and particle size distribution has been estimated. The effect of stirring speed on the mean particle diameter has been studied and it has been observed that particle size increases initially with stirring speed and after achieving the threshold limit, it starts to decrease with increase in stirring speed. The batch filtration experiment has been carried out to estimate the filter cake resistance and it was used to design the continuous belt filter. The drying kinetics has been carried out to estimate the constant drying rate and critical moisture content and these estimated parameters were used for the designing of continuous screw conveyor dryer. The designed continuous belt filter and screw conveyor dryer has been fabricated. The fabricated system has been operated successfully at various flow rates and various particle sizes.

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List of Figures:

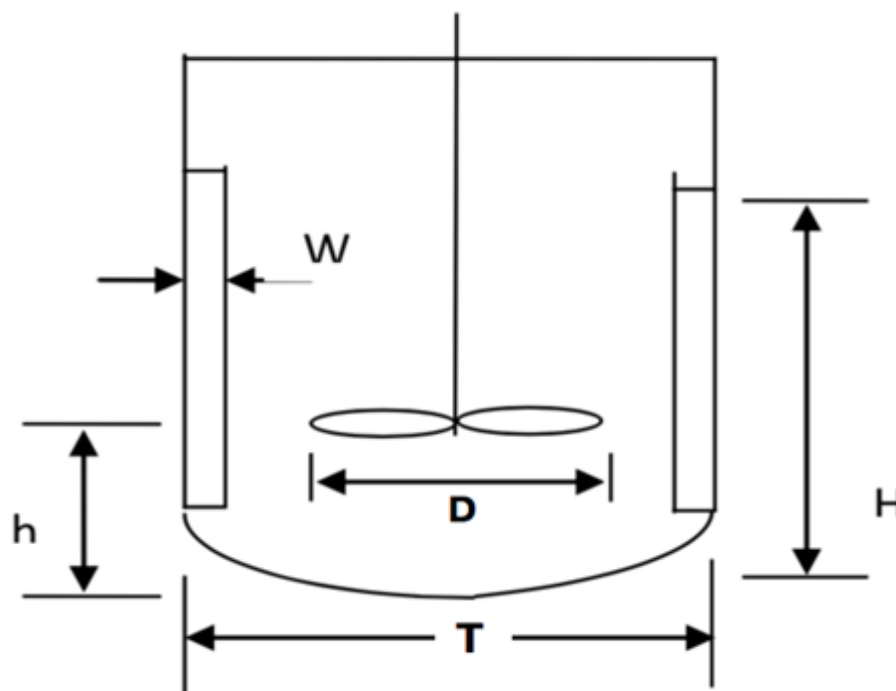


Figure - 1: Batch experimental set-up to manufacture the Calcium oxalate monohydrate

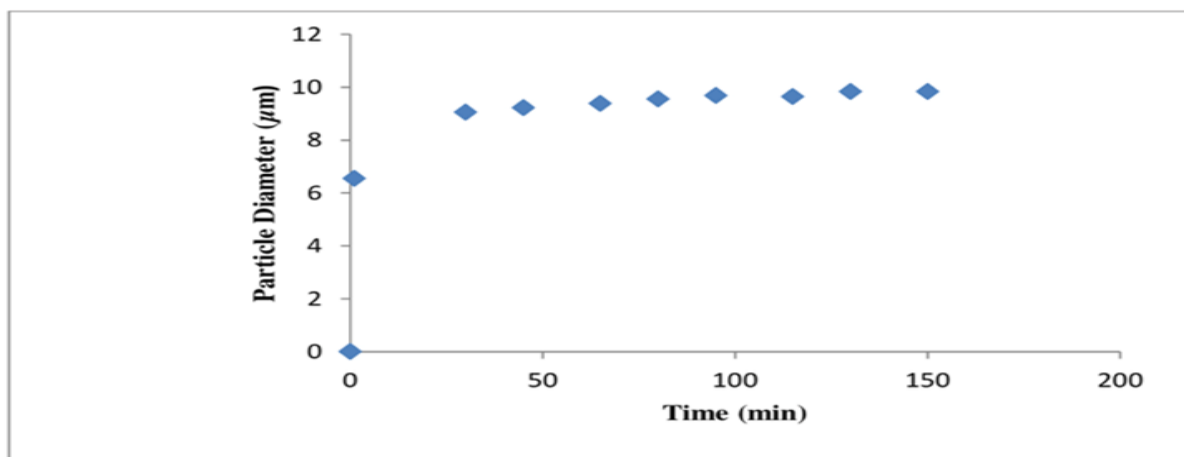
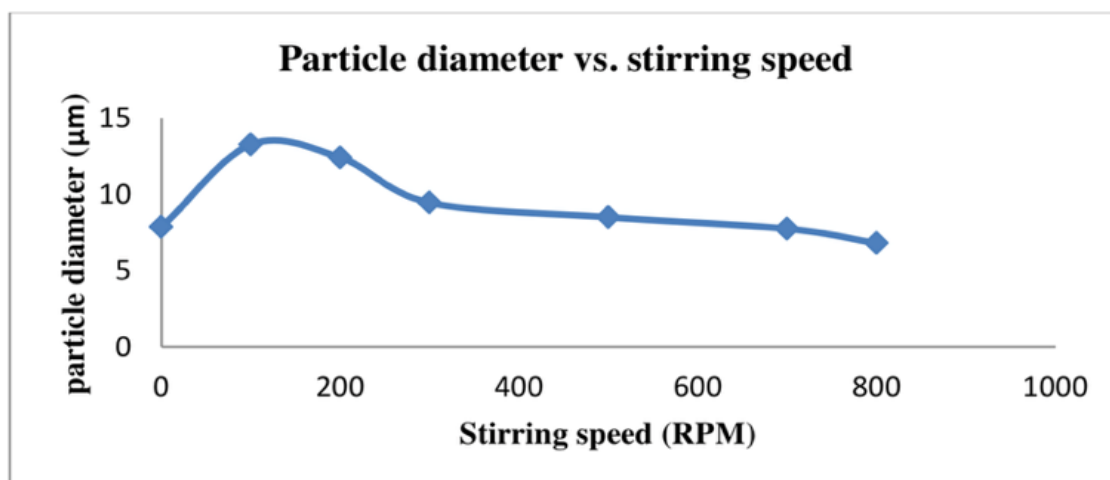
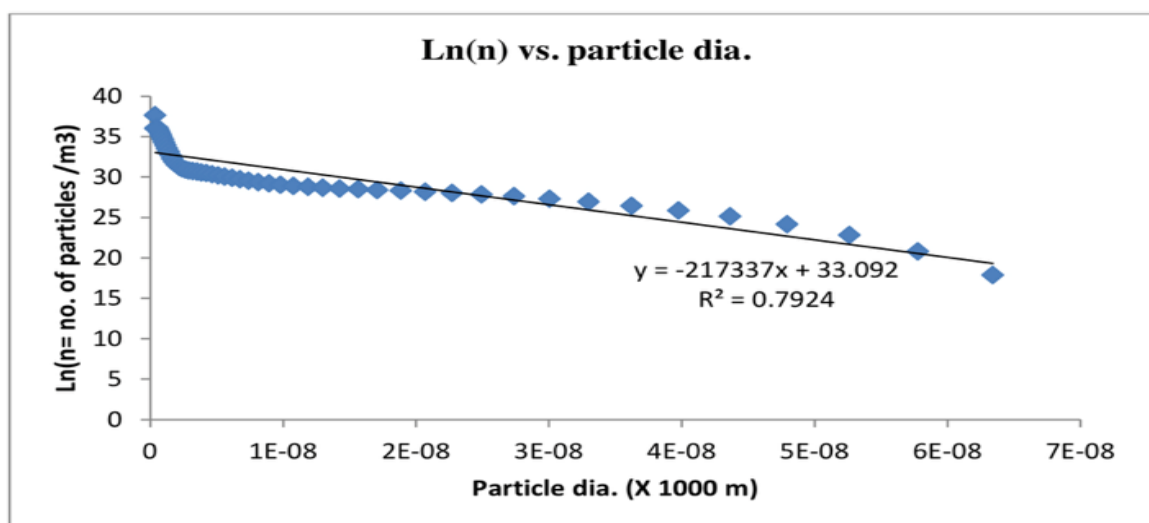
Figure-2 : Variation in mean particle diameter with time.**Figure-3 : Variation in mean particle diameter with speed of stirring****Figure-4 : Particle size distribution.**

Figure-5 : Experimental set-up of batch filtration unit.



Figure-6 : Plot of t/V Vs V .

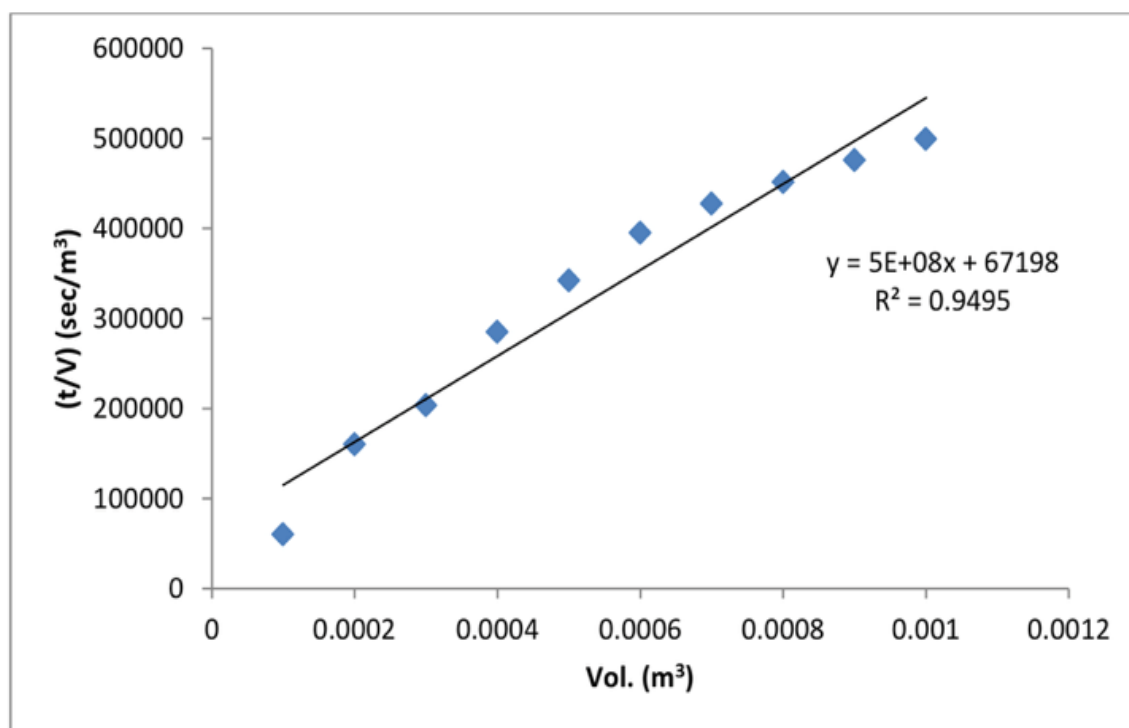


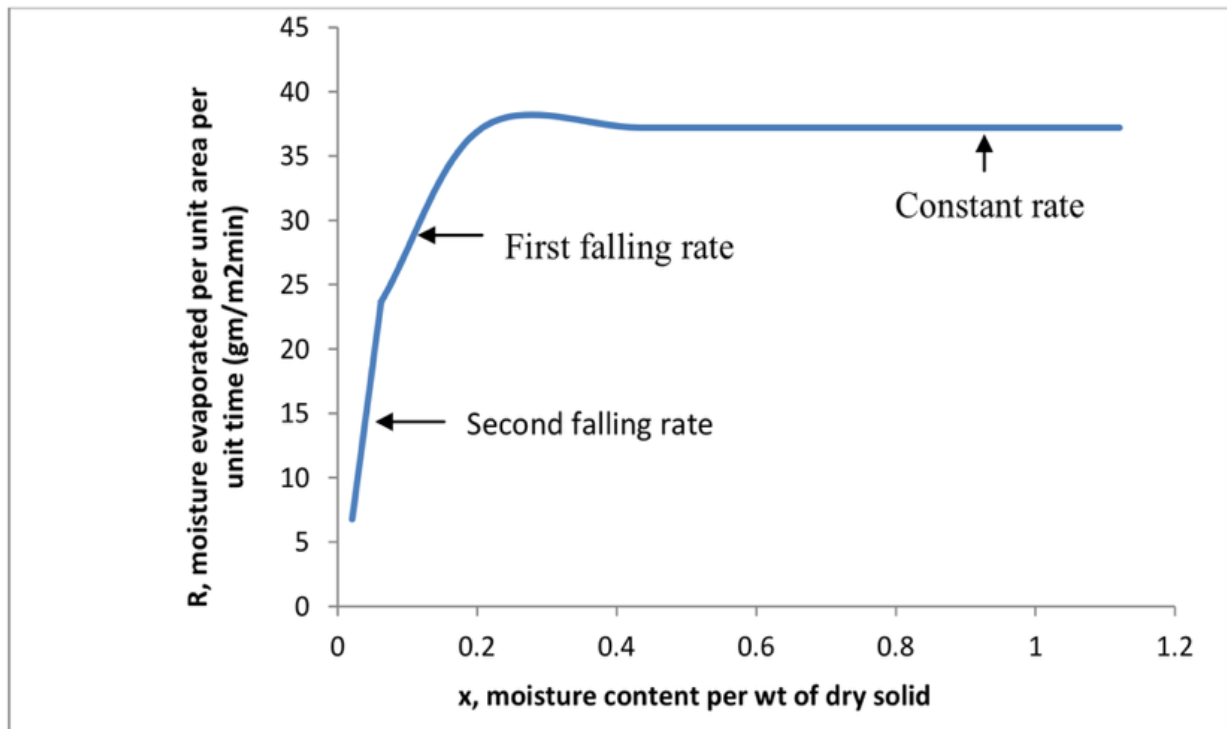
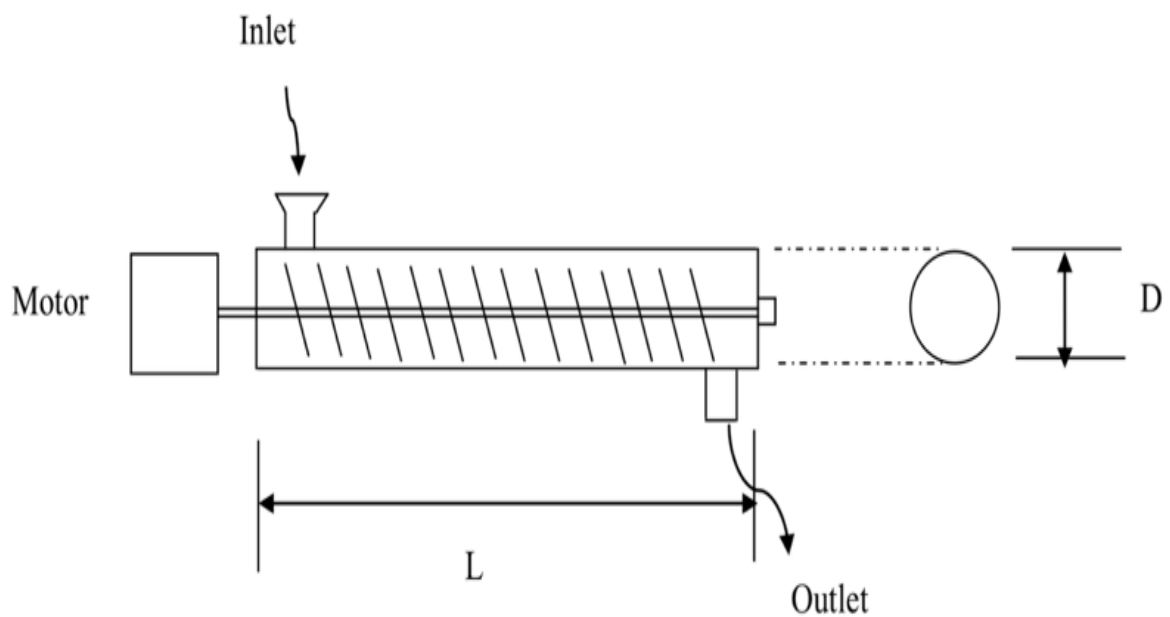
Figure-7 : The graph of drying rate Vs moisture content.**Figure-8 : The schematic of screw conveyor dryer.**

Figure-9 : Cross sectional view of the screw conveyor dryer.

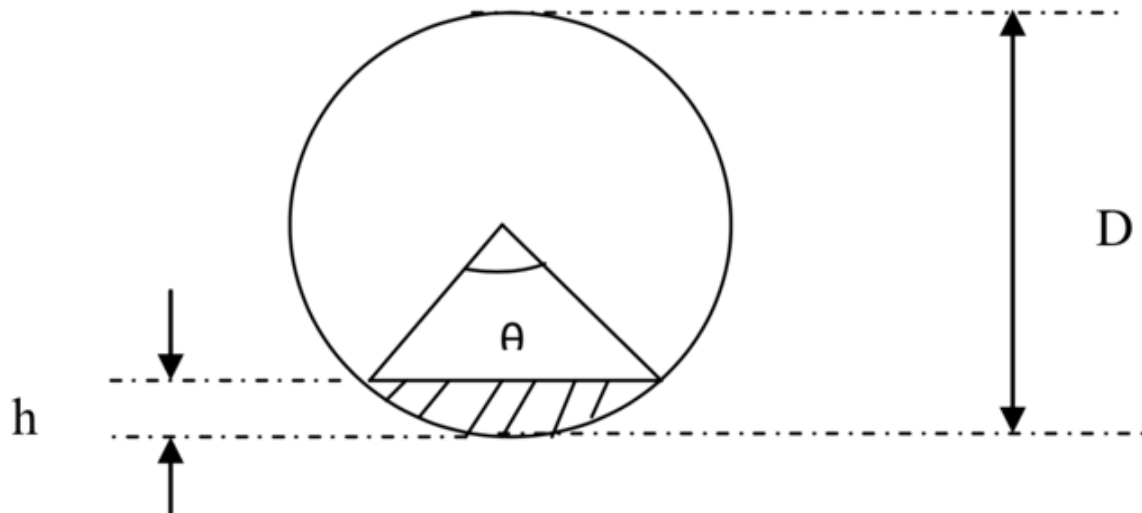


Figure-10 : The schematic of continuous filtration system.

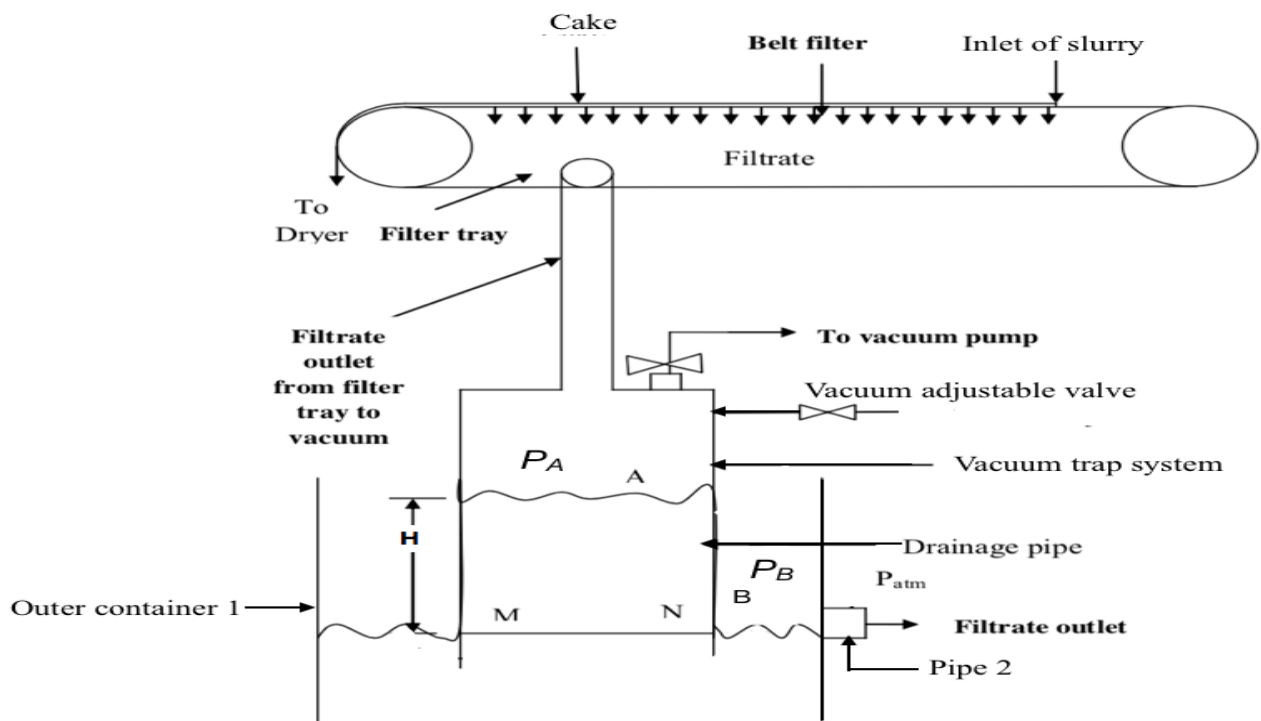
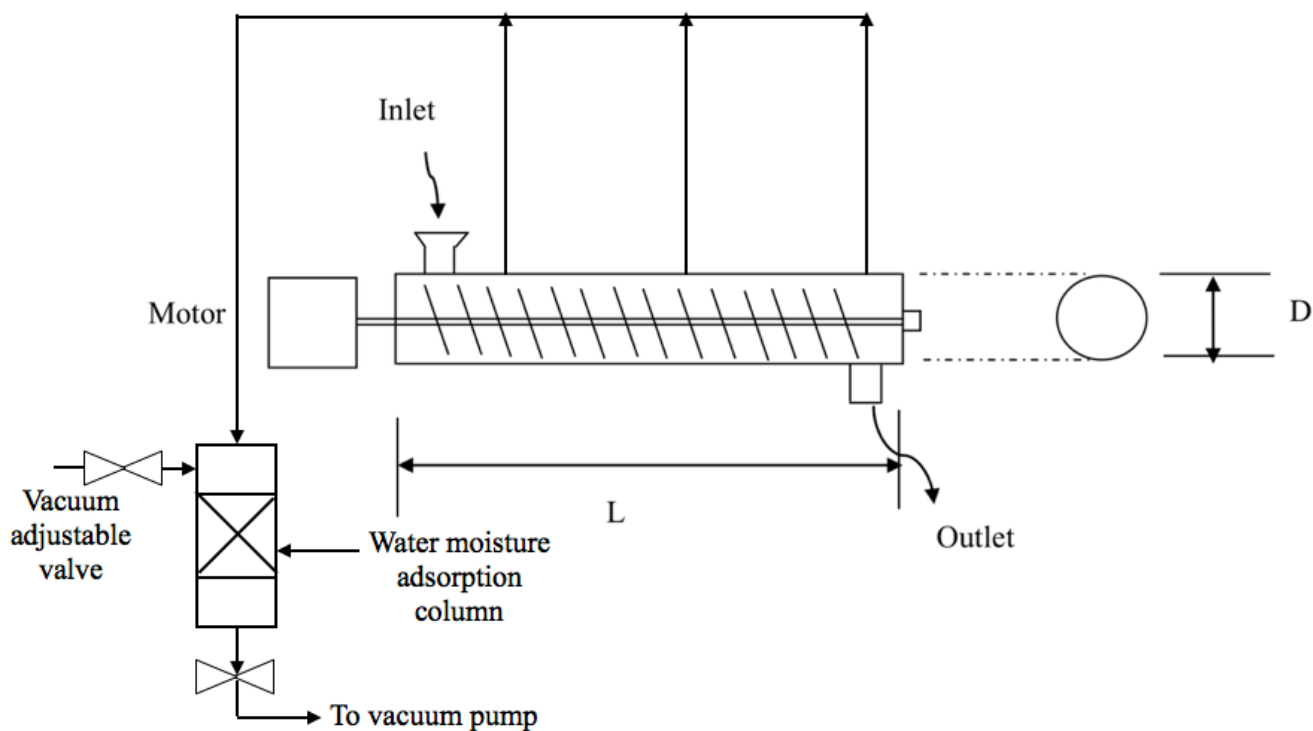


Figure-11 : The schematic of continuous drying system.**Acknowledgement:**

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Author Contributions:

S. G. Shingade and P. D. Gangawane conceived and designed the experiments. S. G. Shingade designed the continuous filtration and drying system and fabricated it as per the designing. S. G. Shingade validated the designed system experimentally. S. G. Shingade and P. D. Gangawane co-wrote the research article.

Competing Interest:

The authors declare no competing financial interest.